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AB Transparent copy paper or diazo material can be prepd. by impregnating paper pulp with a polypropylene resin. Thus, paper from 100% rag is impregnated with a soln. of 30 parts polypropylene (Amopol C 60) in 70 parts naphtha, rolled, stored 1 week, dried below the b.p. of naphtha, moistened with a soln. of 4 ml. ethylene glycol and 0.15 g. saponin in 100 ml. H2O, dried, sensitized with a soln. of 7 g. citric acid, 5 g. thiourea, 2 g. resorcinol, 4 g. 1,4-(Me)(HOCH2CH2)NC6H4N2ZnCl2, and 0.15 g. saponin in 100 ml. H2O, dried, exposed to uv and developed in an NH3 chamber to give a transparent print which can be used as an original for other prints, at 30-60% greater speed than one prepd. from untransparentized paper. Similarly, the transparentized paper can be coated with AgX emulsions, exposed through a negative, developed, and fixed to give a transparent projection positive.

IT Copying paper
Photographic paper
(transparent, contg. propene polymers)

IT 9003-07-0, Propene polymers
(transparent copy and photographic paper contg.)

RN 9003-07-0

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PTO 03-1781

French Patent

Document No. 1399903

Method for Making Transparent the Base Material for Tracing Paper and Intermediary Diazo Paper Using Polypropenes

[Procédé pour rendre transparente la matière de base pour papier à calque et papier diazotype intermédiaire au moyen de polypropènes]

General Aniline & Film Corporation

UNITED STATES PATENT AND TRADEMARK OFFICE

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The present invention relates to a method for making transparent the base material for paper used in tracing papers, in intermediary diazo papers, or certain photo papers that involve soaking the paper using a resin derived from a polymer of a low-molecular-weight olefin.

Tracing papers are generally processed in order to obtain greater transparency to sources of ultraviolet light and to improve the visibility through surface tracings. The base weights of these papers vary according to users' requirements. The improved transparency resulting from the processing enables higher reproduction speeds for industrial drawings and other subjects by the many photocopying machines currently available on the market. The operation that makes transparent such a base material for paper is therefore a major factor in the photocopying field.

Lending transparency to a base material for paper in order to sensitize it using diazo formulas is also extremely important. By making these paper bases transparent, one obtains higher reprinting speeds in "blank printing" machines used to expose and develop diazotype materials. Intermediary diazo

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[1] Numbers in the margin indicate pagination in the foreign text.

papers that are made transparent are generally used to manufacture additional reproductions on other photosensitive materials such as normal diazotype papers, fabrics, films, etc., in blank printing machines.

The types and qualities of raw paper used in tracing papers and sensitized intermediary diazo papers vary considerably, but all of them may be made transparent very advantageously. These papers may consist of 25, 50, or 75% rag paper, with the rest consisting of wood pulp, sulfite pulp for example, or vegetable pulp, bamboo for example; the paper may also consist of 100% sulfite paper. The base weight of these papers may vary, but in most cases 500 sheets of 42.5 X 55 cm paper weigh 5.45 to 9.0 kg.

We have already proposed making transparent the primary material for tracing paper and for intermediary diazo papers by soaking them with a resin such as polystyrene, polymethylstyrenes, and chlorinated aromatic hydrocarbons such as chlorinated diphenyl. Although we obtained some improvement using these materials, the results were far from optimal. Thus, in the case of tracing papers, we note that papers made transparent by prior techniques tend to "shadow" or "veil," which is caused by folding and the breakage of the resin and paper films. The dark lines resulting from this breakage block

ultraviolet light by causing the formation of undesirable lines while making copies on the other reproduction papers. Moreover, the tracing papers and intermediary papers that were made transparent as indicated above show a marked tendency to yellow under the influence of ultraviolet light. Given this fact, it is absolutely necessary to provide a medium that gives transparency without the defects noted above in the papers that have been made transparent.

We observed that when using, as a transparency-giving medium, polypropene alone or mixtures of polypropene with other clear resins such as polyethylene, polystyrene, or polymethyl methacrylate, the base material for tracing paper and intermediary diazo papers was markedly improved over transparent papers currently on the market. Papers made transparent using polypropylene or mixtures with 10 to 50% by weight of other clear resins as indicated above showed a considerable decrease in the "shadow" effect in tracing papers. Moreover, the primary material for the transparent paper discussed herein has excellent heat durability and does not yellow when exposed to sources of ultraviolet light. Additionally, the polypropene resin does not form a hard film, which facilitates plastification of the paper, making it soft and pliable.

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Finally, a major factor in manufacturing transparent papers is the low price of polypropene resin.

Various qualities of polypropene are commercially available. It specific weight varies from approximately 0.89 to 0.91. Usable polymers preferably have an average molecular weight ranging from 600 to 1500. Examples of polypropenes that have proven especially interesting are those sold by "Amoco Chemical Corporation" under the names "Amopol C-60, C-100 and C-175." These liquids have a viscosity ranging from 50 to 200 centistokes measured at 99 degrees C. Their refraction index ranges from 14730 to 14770. The C-60 quality is especially interesting since its molecular weight is the lowest, and it is usable in higher concentrations and at lower viscosity. Lower-viscosity solutions are very interesting thanks to their rapid penetration of the paper and the greater ease with which transparency is obtained. In any case, polypropene resins as a class are especially interesting because they are essentially colorless, odorless, light-stable, they provide a high degree of durability, and they preserve their physical characteristics after prolonged exposure to heat alone or in association with a source of ultraviolet light.

In order to make the primary material for paper transparent, one may proceed in various ways. One may, for

example, dip the paper stock into a polypropene solution in order to ensure complete impregnation, roll the moist paper into a cylinder, and allow the roll of moist paper to sit for a period lasting four days to one week. The paper is then heated in order to dry it while eliminating the solvent.

The paper may also be soaked by applying the polypropene solution onto one of the sides of the paper prior to diazo sensitizing and drying it almost immediately in order to remove the solvent. In this type of method, it is not necessary to roll up the transparent paper or to keep it in roll form while moist, as is recommended in the above-described variation of the method. In this variation of the method, the back of the paper is moistened on the side placed on the wire cloth and is then sensitized by a diazo. In yet another variation, the raw paper is first moistened on the wire cloth side, then sensitized by a diazo on the felt side. We then apply a solution containing a resin made of polypropene or mixtures of the later with the other clear resins mentioned above onto the side of the diazo-sensitized paper facing the wire cloth. The processed paper is then dried at a high temperature in order to obtain a transparent material suitable for producing intermediary diazotype prints. The abovementioned variations on the method

enable use of various installation types to obtain a final product that has the desired degree of transparency.

If the paper must be used to manufacture tracing paper, it is, of course, not moistened or sensitized. Rather, for tracing paper, it is converted into sheets or rolls as needed.

If desired, one may add one or several dyes to the solution in order to make the base paper transparent so as to tint it. These dyes are of the oil-soluble variety, and must be soluble in and compatible with the solvents used to dissolve the polypropene. Among the dyes considered appropriate, we may mention "Alizarine Irisol N Powder Oil Soluble Dye" and "Heliogen Blue Oil Soluble Dye," both of which are commercially available and sold by General Dyestuff Corporation.

The following examples illustrate the invention but are in no way limiting.

Example 1. - We dissolved 30 parts of "Amopol C 60" polypropene in 70 parts by volume of naphtha solvent. We dipped 100% rag paper into the solution until the paper was fully soaked. We then rolled up the moist paper and allowed it to sit for a one-week period. We dried the paper by heating it to above the boiling point of the naphtha solvent. The paper could then be used for diazo sensitizing or directly as tracing paper.

We applied a formula for dorsal damping on the wire cloth side using the following solution, and dried the paper.

Water	100 ml
Ethylene glycol	4 ml
Saponin	0.15 g

We then sensitized the processed paper on the felt side using the sensitizing formula in sepia color and dried it.

Water	100 ml
Citric acid	7 g
Thiourea	5 g
Resorcin	2 g
4-(N-methyl-N-hydroxyethyl) -aminobenzene-diazonium chlorozincate	4 g
Saponin	0.15

Following conversion, the paper is ready for use as an intermediary paper, after exposure to a source of ultraviolet light under a pattern in a blank printing machine and developing it by passing it through an ammonia chamber of the machine in order to make a copy of the model. The transparent prints can then be used as diazo originals in order to make other high-speed reproductions on a normal diazo paper that is more opaque. The method that makes this example transparent enables a

reproduction speed that is 30 to 60% higher than what is obtained with a paper that has not been made transparent.

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Example 2. - We dissolved 30 parts by volume of polypropene in 70 parts per volume of toluene. Into this solution, we immersed 100% sulfite pulp paper to ensure that it was fully impregnated. We then rolled up the paper and allowed it to sit for a period of seven days. We then dried the paper in order to eliminate the toluene. The paper may be used directly as tracing paper or may be sensitized as a diazo intermediary.

Example 3. - We dipped 25% rag/75% sulfite pulp paper into the following solution:

Xylene	70 ml
C-60 type polypropene	30 ml

until the paper was fully soaked in the solution. We then allowed the paper to sit for a one-week period, after which it was dried by heating it to above the boiling point of the solvent. We then applied a dorsal damping formula as in Example 1 onto the side of the paper facing the wire cloth and dried it. The processed paper was then sensitized on the felt side using the following sensitizing formula, and dried.

Water	100 ml
Citric acid	4 g

Boric acid	2 g
Thiourea	5 g
2,2',4,4'-tetrahydroxydiphenyl	2 g
Double salt of zinc chloride and p-diazodiethyl-aniline	3 g

The sensitized paper can be used in the same way as in Example 1.

Example 4. - We coated a transparent paper that was prepared as described in Example 1 using a high-contrast silver halide emulsion. After exposure under a negative, the paper was processed as usual using a black-and-white revealing solution containing a silver halide developing agent, the development was abruptly halted, followed by fixing, washing and drying; we obtained a positive print with excellent picture quality and high transparency that could be used for projection or reproduction.

The following examples show the mixture of polypropene with other resins in order to obtain a suitable transparent paper.

Example 5.

Toluene	70 ml
C-60 polypropene	15 ml
PS-2 polystyrene	15 g

A paper soaked in this solution yielded prints with excellent transparency after coating using a sensitizing diazotype solution as described in Example 1. PS-2 polystyrene is a commercially-available polystyrene resin made by Dow Chemical Company.

Example 6.

Toluene	70 ml
C-60 polypropene	15 ml
Dow V-2 resin	15 ml

A paper soaked in this solution and coated with a sensitizing diazotype solution as described in Example 1 yielded prints with excellent transparency. The Dow V-2 resin is a commercially-available resin sold by Dow Chemical Company and is derived from betamethylstyrene.

Of course, the critical characteristic of the invention is the use of polypropene for making transparent the pulp that is to be used in manufacturing tracing papers or diazo intermediary papers. Polypropene may be used with a small quantity, 5 to 10% for example, of a compatible colorless resin such as polystyrene, if desired.

When the paper is to be used in making intermediary diazo papers, one may use any of the usual diazos. For example, one may use the diazo derived from N,N-diethyl-2-ethoxy-p-phenylene-

diamine, from N-ethyl-2-methyl-p-phenylene-diamine, from N,N-bis(betahydroxyethyl)-p-phenylene-diamine: the diazo compounds described in US Patent No. 2,298,444 published on October 13th, 1942 may be substituted in positions 2 and 5 of the benzene ring by ethoxy, propoxy, butoxy, etc. groups.

What was stated concerning diazos also applies to the coupling compounds. Thus, in addition to the coupling agents cited in the examples, one may use 2,5-xylenol, 2,3-dihydroxynaphthalene, 1,8-dihydroxynaphthalene, resorcin, octyl resorcin, p-methyl-N-phenyl-pyrazolone, beta resorcylic acid, beta resorcylic acid amide, 2-hydroxynaphthalene-3,6-disulfonic acid, H acid, acetyl-acetanilide, or 2,3-dihydroxynaphthalene-6-sulfonic acid. In short, the sensitizing formula and the dorsal damping formula are standard.

The quantity of polymer relative to the solvent varies depending upon the weight of the paper and the desired degree of transparency. These factors are easily evaluated by a technician.

The present invention may be the subject of many variations that are obvious to the expert. For example, the transparent paper is also suitable for use in the silver-salt diffusion transfer method after coating with a colloid layer, such as a

gelatin or polyvinyl alcohol layer containing reduction nuclei such as silver, silver sulfide, etc., in colloidal suspension.

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SUMMARY

The object of the invention is:

1. A method for making transparent a primary material for tracing paper and photosensitive reproduction paper that consists of soaking the paper by means of a solution in an organic solvent of a resin formed essentially of polypropene, keeping the paper in the printed state until it is thoroughly impregnated, then drying it by eliminating the solvent.

2. In this type of method, the additional characteristics that follow, considered alone or in their various technically-possible combinations:

a. The solvent is naphtha, xylene, or toluene solvent;

b. Thorough impregnation is obtained by rolling up the moist paper and allowing it to sit for a period of four days to one week;

c. The transparent paper is damped on the back to prevent it from curling, then is sensitized using a diazo sensitizing solution;

d. The transparent paper is covered in a silver halide emulsion;

e. The paper contains 25, 50, 75, or 100% rag or 100% sulfite pulp.

3. The novel industrial product constituted by a primary material made transparent for tracing paper or for photosensitive reproduction paper, thoroughly impregnated by a resin essentially made of polypropene.

4. In such a product, the following additional characteristics, considered alone or in all of their technically-possible combinations:

a. The primary material is sensitized using a diazo solution to produce a transparent intermediary diazo paper;

b. The transparent paper carries a silver halide emulsion.